A

Synopsis

On

"Synthesis and Characterization of Nickel-Zinc Nano Ferrite Substituting with Chromium by Sol-Gel Technique for Energy Storage Device"

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SYNOPSIS

"Synthesis and Characterization of Nickel-Zinc Nano Ferrite Substituting with Chromium by Sol-Gel Technique for Energy Storage Device"

ABSTRACT:-

A simple, effective and low-temperature technique is used to investigate the effect of rare earth element Chromium substituted in Ni-Zn nanoferrite as (A)x (B)y Fe_2 O_4 (where A is a combination of Ni-Zn metal nitrate and B is rare earth metal (Chromium) nitrate for first series: $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (x = 0,0.1,0.2,0.3,0.4,0.5,0.6, 1 and y=0) and second series: $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (y=0.2,0.4,0.6,0.8,1 and x is optimized from the first series). All the metals used for synthesis were analytical grade in nitrate form. The synthesized samples were characterized by TGA/DTA, FT-IR, XRD, UV-visible, SEM, TEM, EDAX, electrical and dielectric properties. The X-ray diffraction patterns of all the samples provided the information about spinel cubic structure. Also the micro strain, porosity, particle size and lattice parameter were determine by XRD. The chemical composition of the samples was confirmed by energy dispersive X-ray analysis (EDAX). The SEM and TEM study provided information about surface morphology of particles and their uniform particle distribution. The SAED pattern represented the spinel nature of the resultant ferrite nanoparticles. The Optical properties were studied using UV-Visible spectroscopy to calculated band gap energy of the prepared samples. UV-visible spectroscopy was used to obtain band gap energy of the samples. The FT-IR spectra showed two strong absorption bands, which confirmed the presence of M-O stretching band of ferrites. The magnetic properties of the synthesized samples were investigated using VSM/Hysteresis Loop tracer at room temperature. The AC electrical resistivity was measured using LCR-Q. DC electrical resistivity was measured using two probe method. The electrochemical study like, cyclic voltammetry (CV), galvanostatic charge discharge (GCD), Electrochemical Impedance Spectroscopy (EIS) for supercapacitor application

The research work presented in the thesis indicates the effect of Chromium substitution in Ni-Zn for enhancing the properties of synthesized nano ferrite materials. The synthesized ferrite materials have wide applications in several areas such as high frequency electronic devices, technology, sensors, magnetic core, energy storage, microwave device, and electromagnetic interference.

1. INTRODUCTION:

The investigators focus their attention on nanotechnology due to its promising results for enhancing the materials properties. Nanotechnology is the technology of making things small, light and cheap. It is the study of manipulating matter on an atomic and molecular scale which deals with structures of size between 1 to 100 nm. It also involves developing materials or devices possessing at least one dimension within this size. Quantum mechanical effects are very vital at this scale which is in the quantum area. Nanotechnology is very diverse, ranging from extensions of conventional device to completely new approaches based upon molecular self-assembly. It may be able to create many new materials and devices with a vast range of applications such as in medicine, electronics, biomaterials, energy production, and consumer and cosmetic products. At atomic scale, the products show improvements in mechanical properties such as strength, toughness, speed, efficiency, quality and finally cost [1]. Nanoparticles show that larger surface area to volume ratio change important properties like structural, catalytic, electronic and magnetic properties. This change in properties provides the wide scope of their application [2].

Ferrites are electrically non-conductive ferrimagnetic ceramic compound materials which consist of various mixtures of iron oxides such as Hematite (Fe_2O_3) or Magnetite (Fe_3O_4) and other metals oxides. Ferrites are predominantly ionic and have very stable crystal structure which contains iron oxides as major constituents such as Cr, Mn and other elements [3]. A group of materials like cubic spinel ferrites having many applications from microwave to radio frequencies. The general form of spinal ferrite is MFe_2O_4, where M represents a divalent metal ion like Mn, Fe, Mg, Co, Cu, Ni, and Zn [4]. The physical and chemical properties of spinel ferrites depend on method of preparation, cation distribution, dopant concentration, processing conditions, sintering temperature, chemical composition, and types of additives [5-8]. Ferrite nanoparticles have attracted an increasing interest because of their scientific

aspects and applications in permanent magnets, targeted drug delivery, and high density information storage devices [9]. Magnesium ferrite cores can be used as gas sensors, catalysts [10], humidity sensors [11]. Also other ferrites materials can be used in electronic inductors, electro-optic modulators, hydroelectric detector, piezoelectric sensors, electronic sensors, high quality filters, transformer cores, ferrite isolators, memory core industry, multiplayer chip inductor, rod antennas, radio frequency circuits, and in computers, recording devices, magnetic cards and tapes [12]. Many physical and chemical methods are available for the synthesis of ferrite nanoparticles but sol-gel technique is gained the attention of scientist or researcher due to great advantages such as production of nanoparticles with best chemical homogeneity, very small in size, and nano size distribution [13].

Different types of rare earths doped nano ferrite materials have been reported in the literature.

M.K. Shobana et al. studied the yttrium doped cobalt ferrite by using sol-gel autocombustion technique and the structural characterization results the formation of a pure single phase and nanosize crystallites in the range 20 to 70 nm [14]. C.B. Kolekar et al. examined the effect of the substitution of Gd^{3+} on Cu-Cd ferrite using sol-gel autocombustion technique to improve their structural, magnetic, electrical proprieties and thermoelectric power of ferrite [15]. S. Ounnunkad et al. reported the effect of Cr^{3+} substitution on barium ferrite using nitrate-citrate sol-gel autocombustion and concluded that the saturation magnetization decreases, coercivity increases with increasing Cr concentrations [16]. Nandu Chaure et al. investigated Al^{3+} substituted Mg-Ni-Zn ferrite prepared by sol-gel method and study the magnetic properties with magnetization saturation (Ms = 38.7 emu/g), coercivity (Hc = 7 Oe) and initial permeability (μ = 83.7) [17]. Xiaoyan Tan et al. synthesized nanocrystalline $Ni_{0.5}Cu_{0.5}Fe_2O_4$ using sol-gel method and the crystalline structure investigated the single cubic spinel when the precursor was decomposed at 800-1000 ^{0}C [18]. A.A. Sattar et al. observed the transport properties of trivalent substituted La^{3+} ferrites and reported thermoelectric power decreases with increasing in ionic radius [19]. B. Suryanarayana et al. prepared Ni-Cu-Zn ferrite nanocrystals using sol-gel method and study magnetic properties. The typical hysteresis curve provides influence of dopant Cu concentration with density 94% and DC resistivity is increases with concentration [20, 21]. S. Singhal et al. reported effect of Co substituted Ni-Cr

ferrites synthesized using sol-gel autocombution method on optical structural and magnetic properties of ferrites. The observation provided as annealing temperature increases particle size increase up to 45 nm and band gap increase up to 3 eV [22]. S.A. Mazen et al. studied the structural and elastic properties of Li-Ni ferrite and determined the force constants, elastic moduli. They reported that the variation of elastic moduli with composition has been interpreted in terms of binding forces between the atoms of spinel lattice [23]. M.B. Mohamed et al. explored the structural, elastic, and magnetic properties for Al substituted Mn-Zn ferrite prepared by sol-gel method. The elastic moduli were estimated and showed a clear improvement with Al concentration in accordance with the decrease of the bonds lengths [24]. S.A. Mazen et al. studied dielectric behavior of Li-Mn ferrite and reported it is due to effect of polarization process on the reduction of the field inside the medium. Also they determined Debye temperature and elastic moduli which depend on dopant concentration [25]. P.P. Khirade et al. observed the effect of Fe substituted $BaTiO_3$ nanoceramics synthesized by sol-gel autocombution method on phase transformation and observed the dielectric properties at lower frequency [26]. The research work presented by K.K. Bamzai et al. indicated the effect of Ca substitution on magnesium ferrite using ceramic technique and concluded that the saturation magnetization decreases, coercivity increases with increasing Ca concentrations [27].

Despite of many other attempts, there is no specific report on the trivalent substitution of Yttrium doped Nickel, Magnesium, Cadmium ferrite nano powders to the best of our knowledge. In brief survey, it is found that few researchers have worked on this ferrite material. Therefore, we have decided to investigate the structural, magnetic and optical properties of Yttrium doped Nickel, Magnesium, and Cadmium ferrite by simplest sol-gel autocombustion technique. Also, it is important to study the Physics at low dimensions and it has potential of applications in the ferrite based devices.

2. OBJECTIVES OF PRESENT RESEARCH WORK:

The objectives of present research work are describe below:

- To Design and Develop the synthesis technique.
- To select the simplest, sophisticated and effective technique for synthesis of nanoferrite materials. The sol-gel autocombustion method is used to investigate the properties of Chromium substituted in Ni-Zn nanoferrite.
- To synthesize nanocrystalline $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (x=0,0.1,0.2,0.3,0.4,0.5,0.6,1 and y=0) using analytical grade metal nitrates and to investigate the properties of Chromium substituted Ni-Zn ferrite.
- To synthesize nanocrystalline $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (y=0.2,0.4,0.6,0.8,1 and x is optimized) using analytical grade metal nitrates and to investigate the properties of Chromium substituted Ni-Zn ferrite.
- To study the properties of prepared sample powder using characterization techniques such as
  1) TG-DTA for thermal properties.
  2) XRD, EDAX for structural properties,
  3) SEM and TEM for morphological properties,
  4) FTIR, UV-Visible for optical properties,
  5) VSM for magnetic properties, and
  6) To study the electrical and dielectric properties of the prepared sample
  7) To prepare electrodes of NixZn(1-x)CryFe2-yO4. on the conducting base.
  8) To study the electrochemicals like, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and Electrochemical Impedance Spectroscopy (EIS) for supercapacitor application
- To optimize the composition of Chromium substituted Ni-Zn ferrite.

3. STRUCTURE OF THE THESIS:

The thesis is divided into five chapters:

Chapter I: Introduction

First chapter involves a general introduction of nanotechnology with consequence of nanoscale, historical perspective, synthesis and properties of nanomaterials, applications of nanotechnology, and introduction to ferrites nanoparticles. It summarizes the available literature on ferrite materials, advanced techniques of

synthesis, characterization, application etc. This chapter also integrates objectives of research and organization of the thesis.

Chapter II: Experimental

The second chapter deals with the synthesis of Chromium substituted Nickel-Zinc ferrite nanoferrite powder. The kinetics of crystallization of chromium-substituted Ni-Zn ferrite nano ferrite powder study to optimize the synthesis conditions. Various history-dependent parameters such as synthesis time, temperature, pH of the gel, sintering temperature, etc also study for optimization.

This chapter also explains the chemical analysis of raw materials, sol-gel auto-combustion synthesis, powder characterization and flow chart for auto-combustion synthesis.

The sol-gel process (auto-combustion techniques) is the conversion of a precursor solution into an inorganic solid via polymerization reaction induced by water. The metal nitrates are most widely used precursor because they react readily with water. The sol-gel process is widely used in the fields of materials science and ceramic engineering for the fabrication of materials starting from a chemical solution (sol), that acts as the precursor for gel and gel gets auto burnt to form the desired product.

Sol-gel auto combustion techniques consist of following steps

1. Preparation of solution by dissolution of inorganic salts (nitrate metals) in water.
2. Conversion of the homogeneous solution into a sol by treatment with a suitable reagent (citric acid).
3. Maintaining the pH as solution by using a liquid (ammonia liquid).
4. By using continuous heat treatment, the formation of gel.
5. Gel gets auto burnt to form the desired product,

Stepwise approach: The following stepwise approach to achieve the objectives stated in the research proposal.

Step-I: Design, development and installation of the basic infrastructure namely sol-gel auto-combustion technique.

Process: Chromium nitrate, Ferric nitrate, Nickel nitrate, Zinc nitrate to use as a raw material in analytical grade, while Citric acid as fuel to prepare composition of $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (x = 0,0.1,0.2,0.3,0.4,0.5,0.6, 1 and y=0) and second series: $Ni_xZn_{(1-}$

$_{x)}Cr_yFe_{2-y}O_4$ (y=0.2,0.4,0.6,0.8,1 and x is optimized from the first series) using the stoichiometric ratio. The distilled water to use as a solvent.

Step-II: To study the characterization of the $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (x = 0,0.1,0.2,0.3,0.4,0.5,0.6,1 and y=0) and second series: $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (y=0.2,0.4,0.6,0.8,1 and x is optimized from the first series) for different compositions. Following techniques were used for the characterization.

i) **Structural Properties:**

X-ray diffraction (XRD), Energy dispersive X-ray spectroscopy (EDAX)

ii) **Morphological Properties:**

Scanning Electron Spectroscopy (SEM), Transmission Electron Microscopy (TEM)

iii) **Magnetic Properties:**

Vibration Sample Magnetometry (VSM)

iv) **Optical Properties:**

UV-Visible Spectroscopy, Fourier Transform Infrared (FT-IR) spectroscopy,

v) **Dielectric Properties:**

Dielectric resistivity/conductivity using LCR-Q,

vi) **Electrical Properties:**

Electrical resistivity/conductivity using two probe methods,

vii) **Thermal Properties:**

Thermo Gravimetric and Differential Thermal Analysis (TG-DTA),

Chapter III: Characterization Techniques

The synthesis materials need to be completely characterized by a number of physical, spectroscopic and electrical techniques to illuminate their structural features, morphology, thermal stability, absorption band gap, surface area, electrical and dielectric behavior, etc. Sintered product as in step-II was characterized by various analytical tools like X-ray diffraction (XRD), Energy dispersive x-ray spectroscopy (EDAX), Fourier transformation infrared (FTIR), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), UV-visible Spectroscopy, Thermo Gravimetric and Differential Thermal (TG/DTA), Vibration Sample Magnetometry (VSM), Two Probe method (Electrical and Dielectric), etc and to use for evaluation of properties.

Chapter IV: Results and Discussion

Numerous experimental studies will be conducted in our research work with Chromium substituting Ni-Zn nano ferrite by sol-gel auto combustion technique.

Chromium substituted Ni-Zn ferrite:

The $Ni_xZn_{(1-x)}Cr_yFe_{2-y}O_4$ (y=0.2,0.4,0.6,0.8,1 and x is optimized) nanoferrites will be synthesis using sol-gel auto-combustion method. The following properties are considered.

- Structural Properties: The XRD analysis nanoparticles crystalline size It gives the information about structure. The EDAX spectrum exhibited the content composition.
- Morphological Properties: The SEM images study the distribution of nanoparticles.
- Magnetic Properties: The magnetic properties study using a hysteresis loop of synthesis sample. The parameters Hc (Coercievity), Mr (Magnetic Remenance), Ms (Magnetic Saturation) and R (Remenance ratio) with Cr^{2+} concentration.
- Optical Properties: The optical properties of prepared samples will also investigate by FT-IR and the UV-visible graph
- Electrical Properties: To study the resistivity and dc conductivity.
- Dielectric Properties: The dielectric properties will be studied using LCR-Q meter
- Thermal Properties: To study the TGA-DTA analysis,
- Electrochemical analysis: cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and Electrochemical Impedance Spectroscopy (EIS) for supercapacitor application

Chapter V: Conclusions and Summary

This chapter presents an overall summary of the work done and describes the major findings of the research work.

The nano ferrites of Chromium are substituted in Ni-Zn at different compositions synthesis. The analysis of thermal property will be discuss by TGA-DTA. The XRD analysis of Chromium substitution in Ni-Zn at different concentration represents its effect on properties. EDAX analysis confirmation the purity and the stoichiometry composition of the samples. The SEM and TEM confirmation of the nanoparticle. TEM analysis reveals the morphology of ferrite samples. The FT-IR spectroscopy confirmation the synthesis samples are ferrites. The UV-visible spectroscopy analysis

the band gap energy of samples. The magnetic property (VSM) of the prepared samples.

The energy storage device analysis will be perform by electrochemical studies, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and Electrochemical Impedance Spectroscopy (EIS).

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PUBLICATIONS:

1) ***R. B. Bhise***, Vaibhav Bhong, S. M. Rathod, "**Influence of Structure and Magnetic Properties of Y^{3+} Doped Ni-Cd Nano ferrite by Sol-gel Autocombustion Method**"**,** ***Bionano Frontier***, Vol. 8(3), Pp. 107-109, December 2015, Print ISSN 0974-0678, Online: 2320-9593, www.bionanofrontier.org
2) ***R. B. Bhise***, S. M. Rathod, "**Preparation of Y^{3+} Substituted Mg-Cd Ferrites by Sol-gel Method: Structural and Magnetic Properties**", ***International Journal of Chemical and Physical Sciences*** (**IJCPS**), Vol. 5(7), Pp. 1-5, FESEM-2016, ISSN:2319-6602, www.ijcps.org
3) ***R. B. Bhise***, S. M. Rathod, "**Synthesis of Nanosized Y^{3+} Doped Ni-Mg-Cd Ferrite Powders and their Structural, Magnetic properties by Sol-gel Autocombustion Method**"**,** ***Research Journal of Material Sciences*****-ISCA,** Vol. 4(5), Pp**.** 1-4, June 2016**,** E-ISSN 2320–6055, www.isca.in , www.isca.me
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8) ***R.B. Bhise,*** S.M. Rathod, **"Optical Behaviour of Yttrium Doped Nickel-Magnesium-Cadmium Ferrite prepared by Sol-gel Autocombustion Method",** ***International Journal of Nanotechnology and Application (IJNA)***,

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ORAL /POSTER PRESENTATION IN SYMPOSIA:

1) "**Influence of Structure and Magnetic Properties of Y^{3+} Doped Ni-Cd Nano ferrite by Sol-gel Autocombustion Method**", **International Conference**- Dec 28-30, 2015 held at Department of Physics, BAM University, Aurangabad.
2) "**Structure and Magnetic Properties of Y^{3+} Doped Mg-Cd Nano ferrite by Sol-gel Autocombustion Method**", **International Conference**- Jan 28-30, 2016 held at Department of Physics, Shivaji University, Kolhapur.
3) "**Preparation of Y^{3+} substituted Mg-Cd Ferrites by Sol-gel Method: Structural and Magnetic Properties**", **International Conference** FESEM-2016 Feb 5-6, 2016 held at Baburaoji Gholap College, Sangavi (Pune).
4) "**Synthesis of Nanosized Y^{3+} Doped Ni-Mg-Cd Ferrite Powders and their Structural, Magnetic properties by Sol-gel Autocombustion Method**", **State level Conference**, Mar 8-9, 2016 held at Hutatma Rajguru College, Rajgurunagar (Pune).
5) **Structural, Electrical and Optical properties of Y^{3+} doped $Ni_{1-x}Cd_xFe_{2-y}O_4$ Spinels Nanoferrite**", **International Conference,** Dec 8-9, 2016 held at Hutatma Rajguru College, Rajgurunagar (Pune).

Research Scholar **Research Guide**

Title of Synopsis	**Synthesis and Characterization of Nickel-Zinc Nano Ferrite Substituting with Chromium by Sol-Gel Technique for Energy Storage Device**
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